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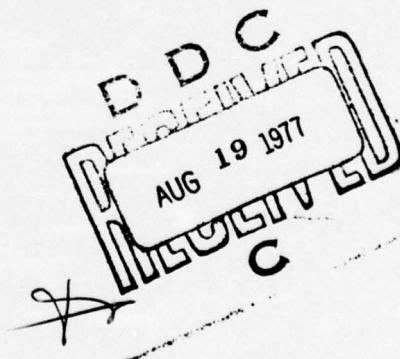
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O.T. Inal and L.E. Murr

June, 1977

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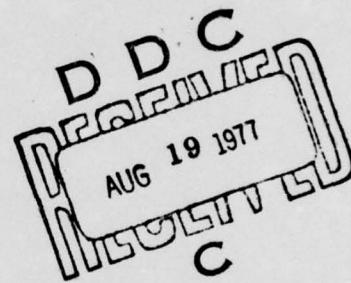
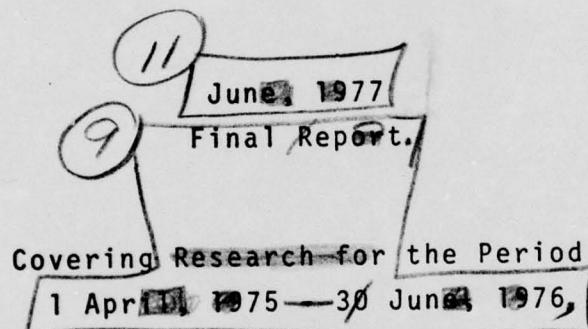
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LASER IRRADIATION OF METALS AND ALLOYS.

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I. INTRODUCTION

The irradiation of metals and alloys by a high-intensity laser beam is known to induce both thermal and shock-stress damage. This damage can occur as surface ablation, the formation of melt or vaporized zones, the formation of fractures, the formation of spall voids on the rear surface of laser-impacted regions, or the formation of defects within the impact zone by the propagating laser-shock pulse. Laser-induced melting and vaporization in beryllium has been demonstrated by direct observations in the scanning electron microscope (1) and Bushnell and McCloskey (2) have previously discussed the promotion and propagation of a stress wave as a result of thermal expansion following the rapid absorption of radiant (laser) energy at the surface of an elastic solid provided the pulse duration is sufficiently short. The production of cracks and laser-induced fracture especially in brittle materials is also well established (3). Metz and Smidt (4) have also concluded that vacancy concentrations estimated to be as large as 1 atomic per cent were induced in nickel and vanadium foils by bombardment with high-intensity laser pulses, and attributed their production to non-conservative motion of jogs on screw dislocations as originally postulated by Kressel and Brown (5) to explain the apparent vacancy production in nickel shock-loaded in the range 70-330 Kilobars.

At the time the present research was proposed, an experiment had been devised to determine the effect of shock loading on the vacancy concentration in elastic solids using the field-ion microscope. The field-ion microscope is

the only means for directly observing single atoms or atomic aggregates; and conversely single vacancies and vacancy clusters. This experiment, described in detail by Murr, et al. (6,7), involved the simultaneous shock loading of molybdenum sheet and wire samples to facilitate examination by both transmission electron and field-ion microscopy techniques.

It was the purpose of this research program to develop a technique for the direct observation of laser-induced damage at the atomic level. This was to be accomplished by laser-irradiation of metal or alloy emission end-forms directly within a field-ion microscope. It was initially thought that such observations could shed some light on the mechanism of laser-induced metal melting, vaporization, sputtering, spallation, or related shock effects, and in particular provide direct evidence for any laser shock phenomena relating to the production of individual vacancies or vacancy clusters as postulated by Metz and Smidt (4), and already demonstrated by field-ion microscopy observations in shock-loaded molybdenum (6,7).

E.W. Müller (8) was the first to attempt to observe laser irradiation effects directly within the field-ion microscope. His observations of what he described as laser damage in tungsten were not at all convincing, and little explanation was offered as to the mode of damage and the mechanism involved. The damage was not very extensive and was apparently not representative of a high-intensity laser pulse.

II. EXPERIMENTAL DESIGNS

It was necessary at the outset to design and construct a special field-ion microscope which would allow metal whisker or filament end-forms to be observed and laser irradiated in-situ. This required a window which would allow a focused laser beam to impinge upon the end form or the filament shank. A thru-port arrangement was designed which would allow the focused laser beam to be viewed and the specimen shank and end form to be aligned. This basic design is illustrated in the schematic of Fig. 1.

The basic field-ion microscope design consisted of a jacket arrangement of heliarc-welded stainless steel cylinders. The basic design utilized flange arrangements for the optical thru-ports and the viewing screen, and is shown reproduced in Fig. 2.

The completed field-ion microscope employed special optical glass cylinders in the laser thru-ports and a channel-plate image intensifier was added to the viewing screen. While the system was devised to operate at liquid helium temperatures, it was envisioned that the bulk of the work could be satisfactorily executed using liquid nitrogen. A standard diffuser consisting of a resistively-heated vycor finger was utilized in the admission of standard helium imaging gas, and special needle valve arrangements were incorporated into the design for the admission of other imaging gases or gas mixtures. The design also incorporated a vapor gettering arrangement utilizing liquid-nitrogen cooling of the getter walls. These

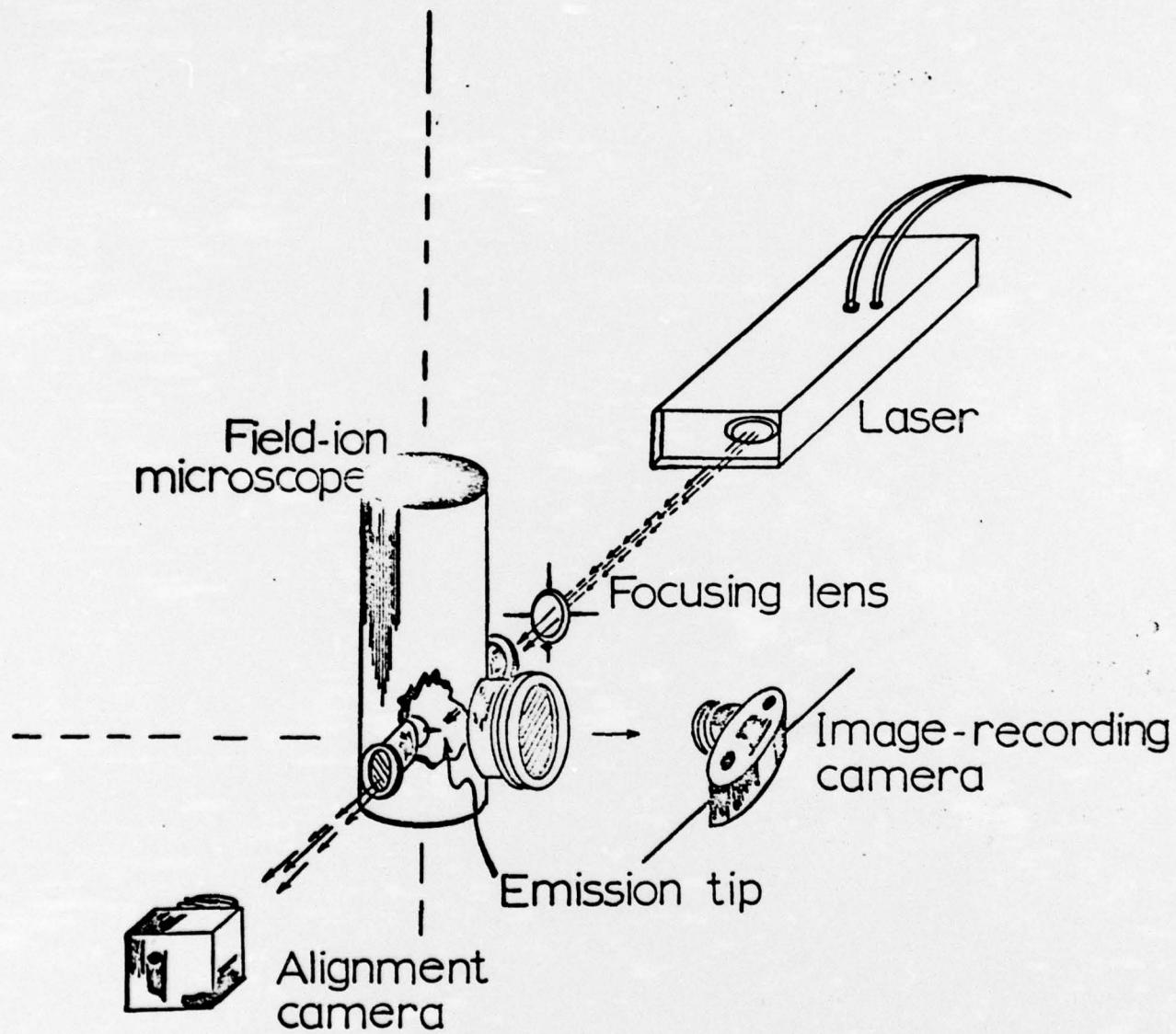


Fig. 1. Field-ion microscope in-situ irradiation system (schematic).

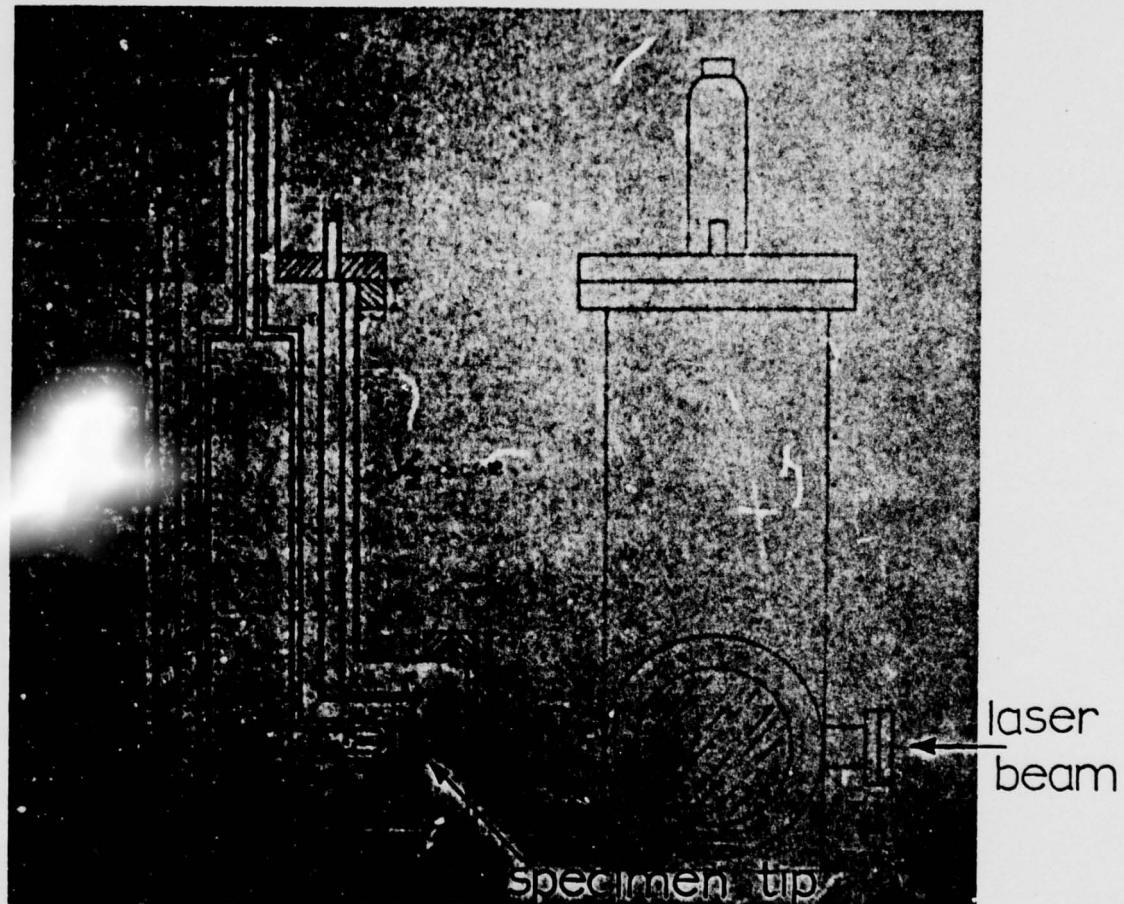


Fig. 2. Basic field-ion microscope construction design.

features are illustrated in Figs. 3-5. Figure 6 shows the principal features of the instrumentation requirements consisting of vacuum monitoring, channel-plate (image intensification) electronics, high-voltage (field ionization) power, and ancillary systems including getter and diffuser power supplies and general system (vacuum, etc.) power supplies.

III. EXPERIMENTAL METHODS

In order to compare the possible laser-induced shock effects with those already identified for explosively-induced shock effects using the transmission electron microscope (6,7,9), especially beryllium (9), thin films of beryllium were prepared and examined in the electron microscope in order to establish a reference for laser-irradiation studies. In addition, small diameter (0.003 in.) wires of beryllium were prepared for observation in the field-ion microscope. The morphology and other features of these wire samples was monitored by observations in the scanning electron microscope. Preliminary alignment experiments were also performed using a small He-Ne laser system which did not have sufficient power to allow any significant laser-irradiation experiments to be conducted.

In preliminary tests with beryllium end forms it was observed that the images were poorly resolved as a result of intrinsic instabilities of the images in the field-ion microscope. It was also observed during the actual testing of the field-ion microscope that explosively-loaded molybdenum wires contained vacancies and vacancy clusters indicative of shock-induced

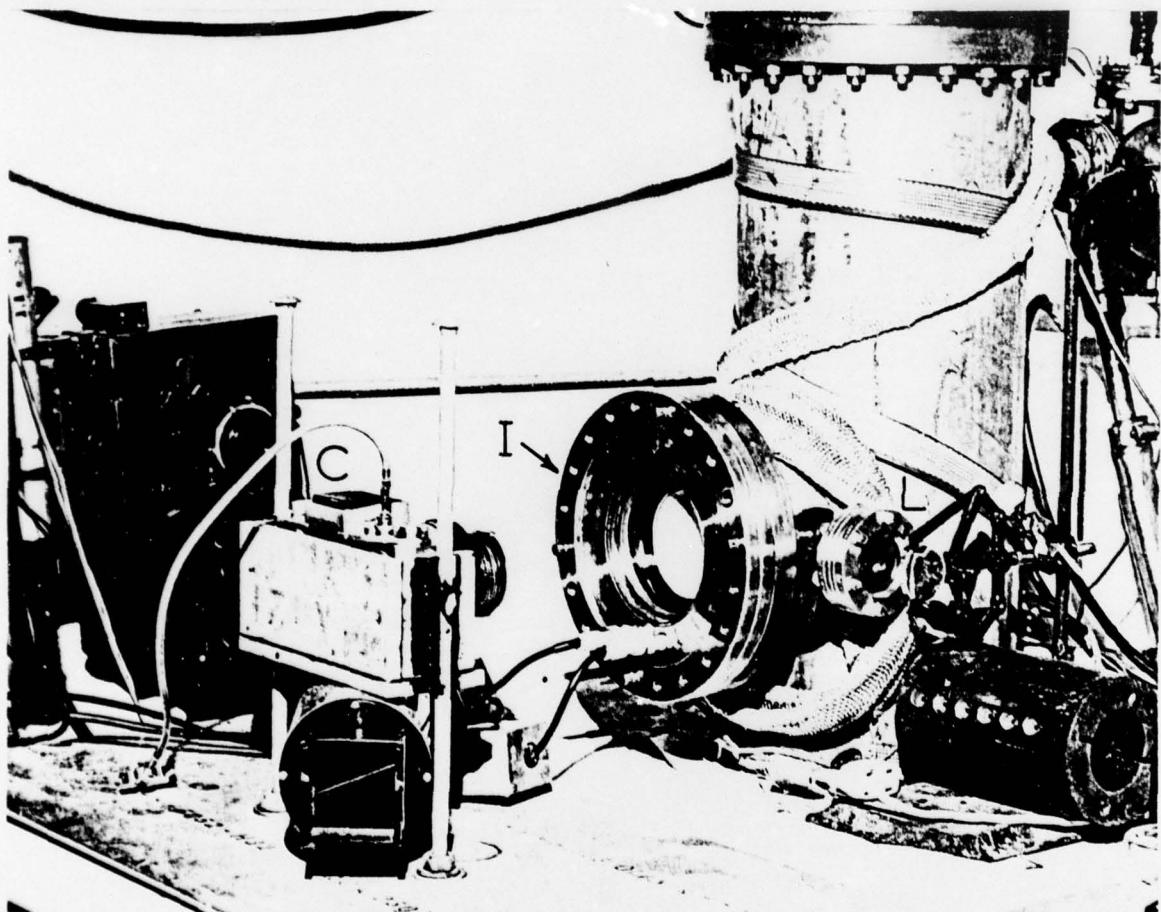


Fig. 3. Field-ion microscope showing channel-plate (imaging screen) (I), laser thru-port (L), and camera (C).

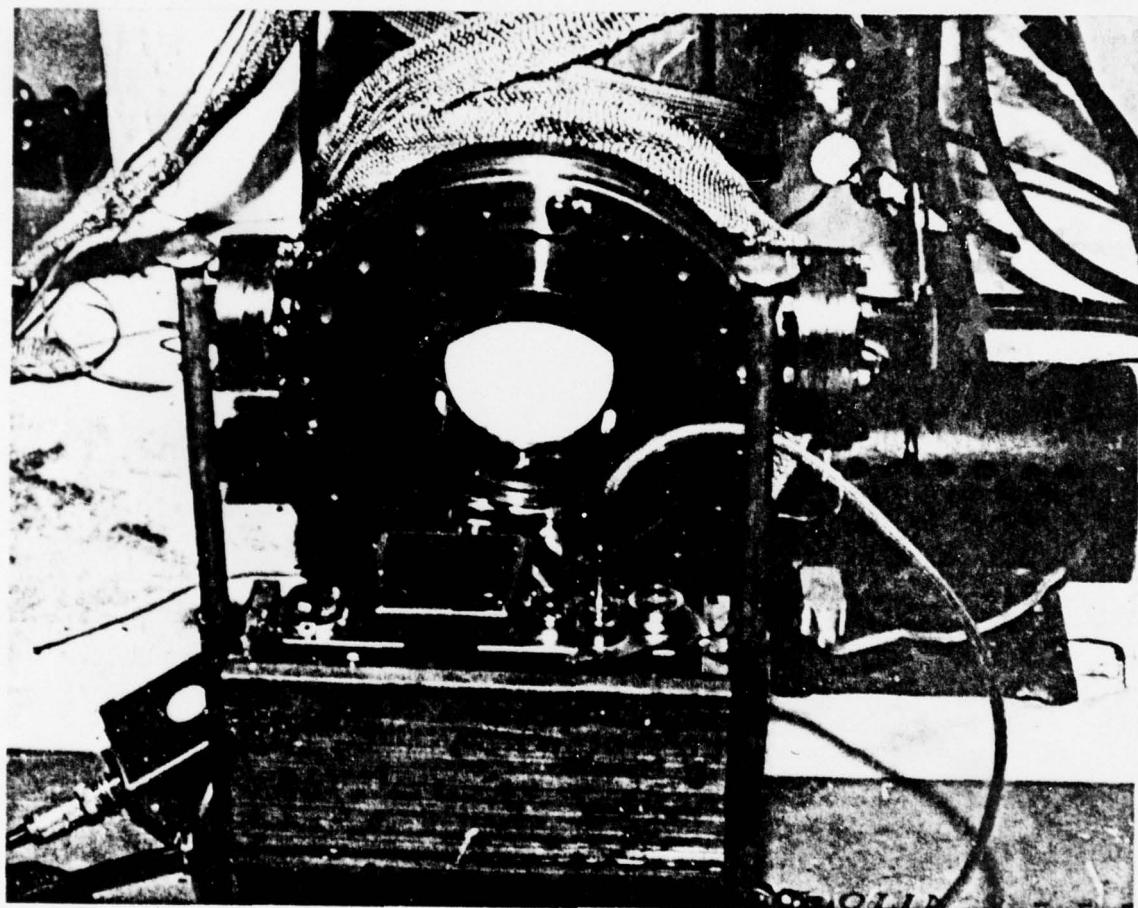


Fig. 4. Field-ion microscope.

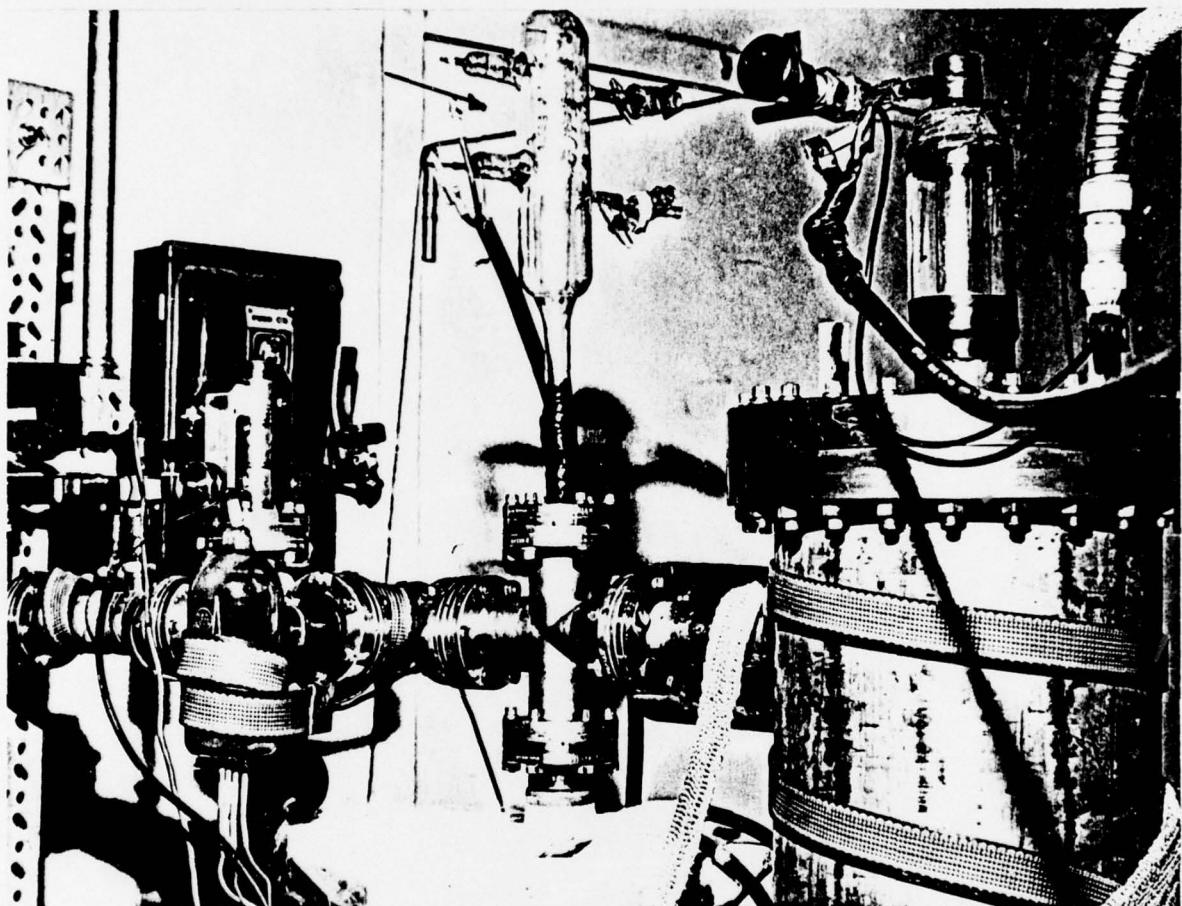


Fig. 5. Image gas (helium) diffuser and vacuum system of special field-ion microscope.

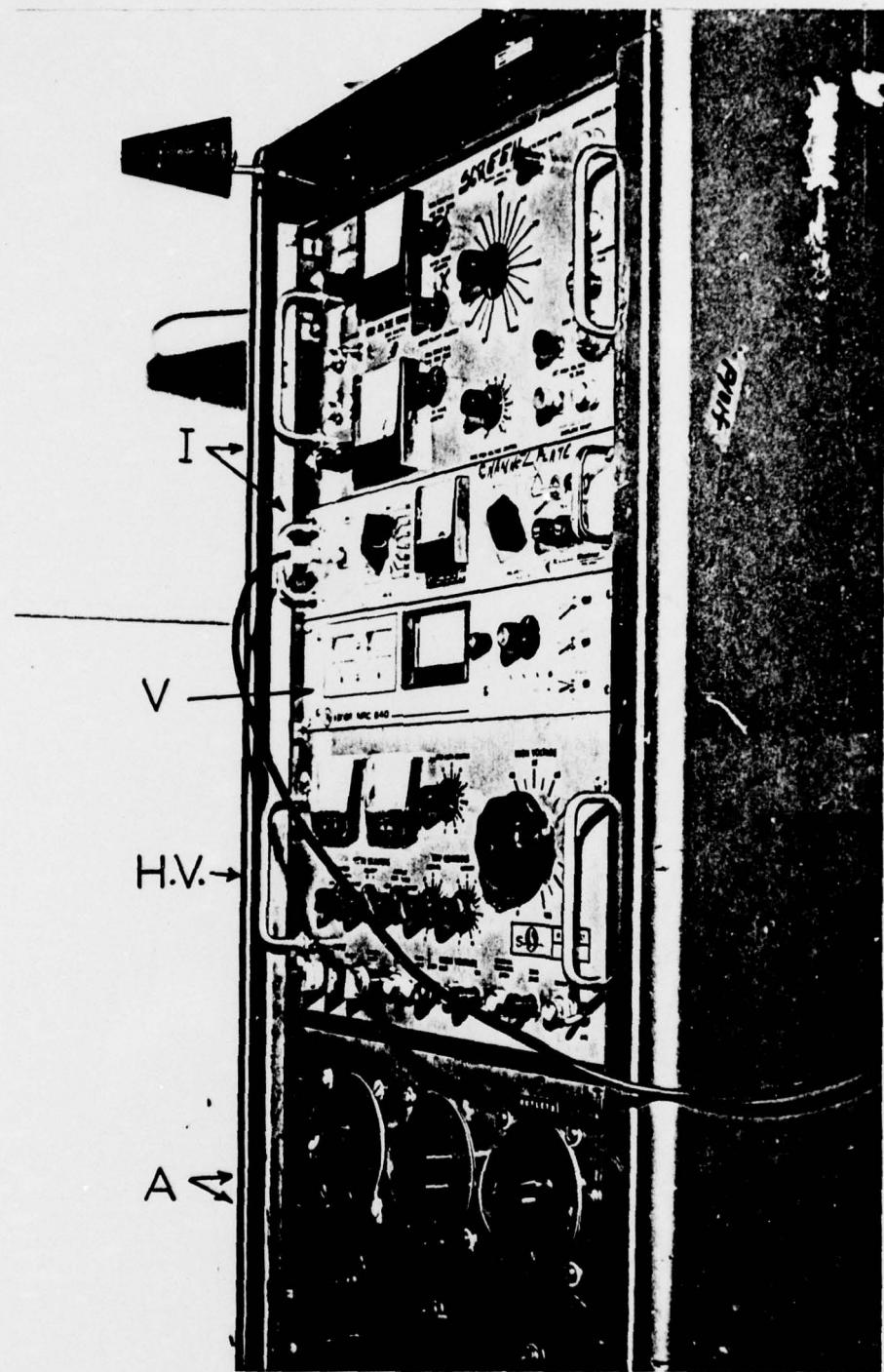


Fig. 6. Instrumentation panel for field-ion microscope showing vacuum monitoring section (V), image intensification (channel-plate) power (I), high-voltage power for field ionization and imaging (H.V.), and ancillary power systems (A).

effects (6,7), and because of the importance of molybdenum as a laser mirror and mirror substrate material, it was considered expedient to attempt to observe and compare laser-irradiated molybdenum with explosively shock-loaded molybdenum, and preliminary studies were also carried out on the observation of molybdenum as well as tungsten end forms.

IV. PRELIMINARY RESULTS AND DISCUSSION

Figure 7 shows the structure typical of beryllium sheet observed in the transmission electron microscope while Figure 8 illustrates the typical morphology of the emission end forms prepared for field-ion microscopy. The emission specimen shown in Fig. 8(a) is shown welded to a loop which allows the tip to be inserted into the field-ion microscope. The views shown in Fig. 8 are essentially those which would be observed on sighting through the laser thru-port on the side of the field-ion microscope as shown in Fig. 3.

As noted previously, attempts to produce good-quality field-ion micrographs of the beryllium were not very successful as a result of the instability of the material in the field-ion environment, possibly as a result of oxygen affinity or other features. The instrument was, however, successfully tested with a number of refractory materials, including molybdenum and tungsten. Typical examples of field-ion images for molybdenum and tungsten are shown in Figs. 9 and 10.

Numerous images of annealed molybdenum and tungsten end forms were obtained during the preliminary testing of the



Fig. 7. Beryllium sheet structure observed in the transmission electron microscope. (a) Small-grained structure of commercially sintered-rolled beryllium sheet. (b) Dislocation networks in annealed sheet Be. (c) Dark-field image showing grain boundary defect structure in annealed Be sheet.

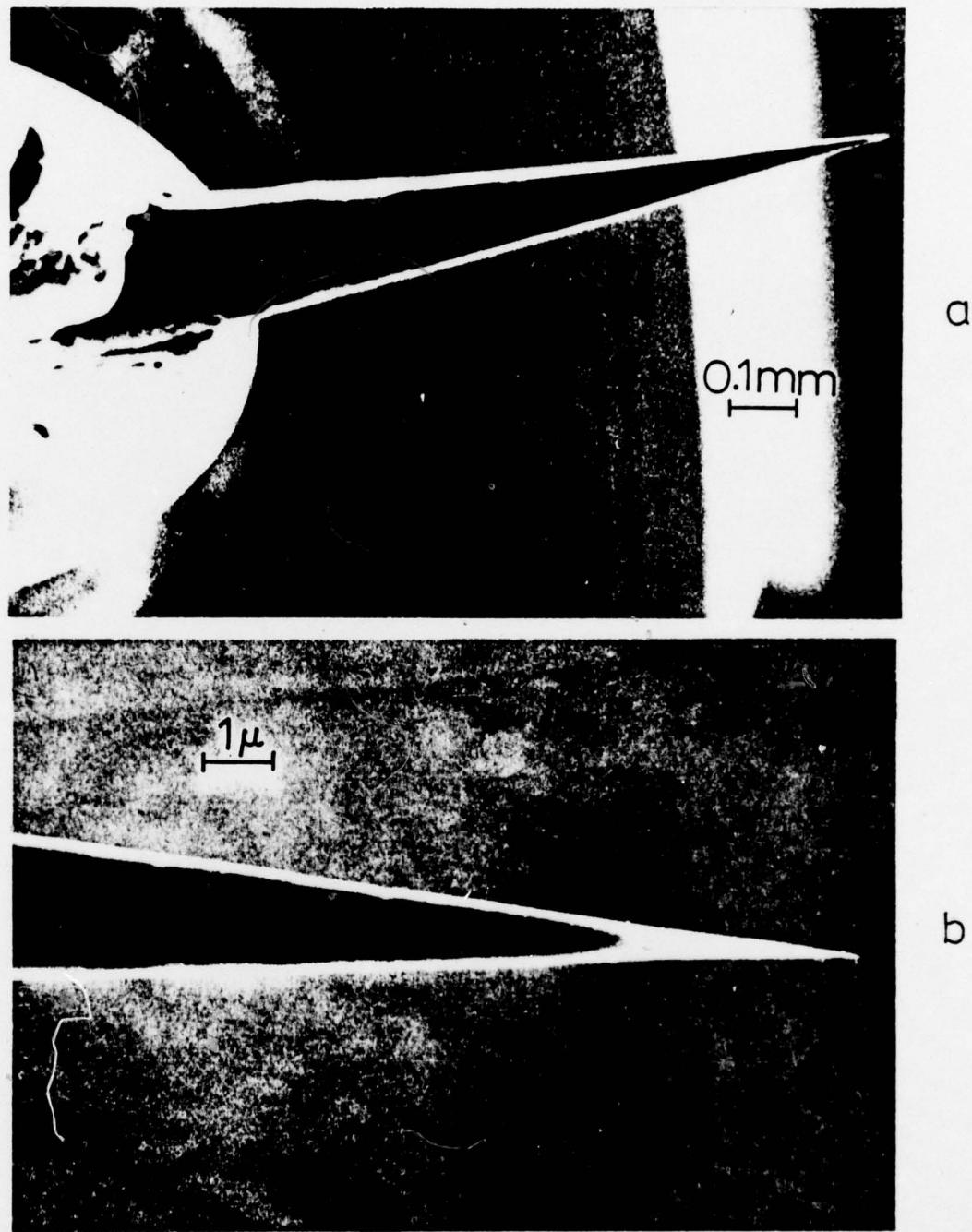


Fig. 8. Beryllium emission end forms observed in the scanning electron microscope. (a) Be emission tip attached to wire loop. (b) Be emission end form showing apparent electron transmissibility at end and edge (side) sections.



Fig. 9. Surface-atom image of molybdenum observed in the field-ion microscope using liquid-helium cooling.

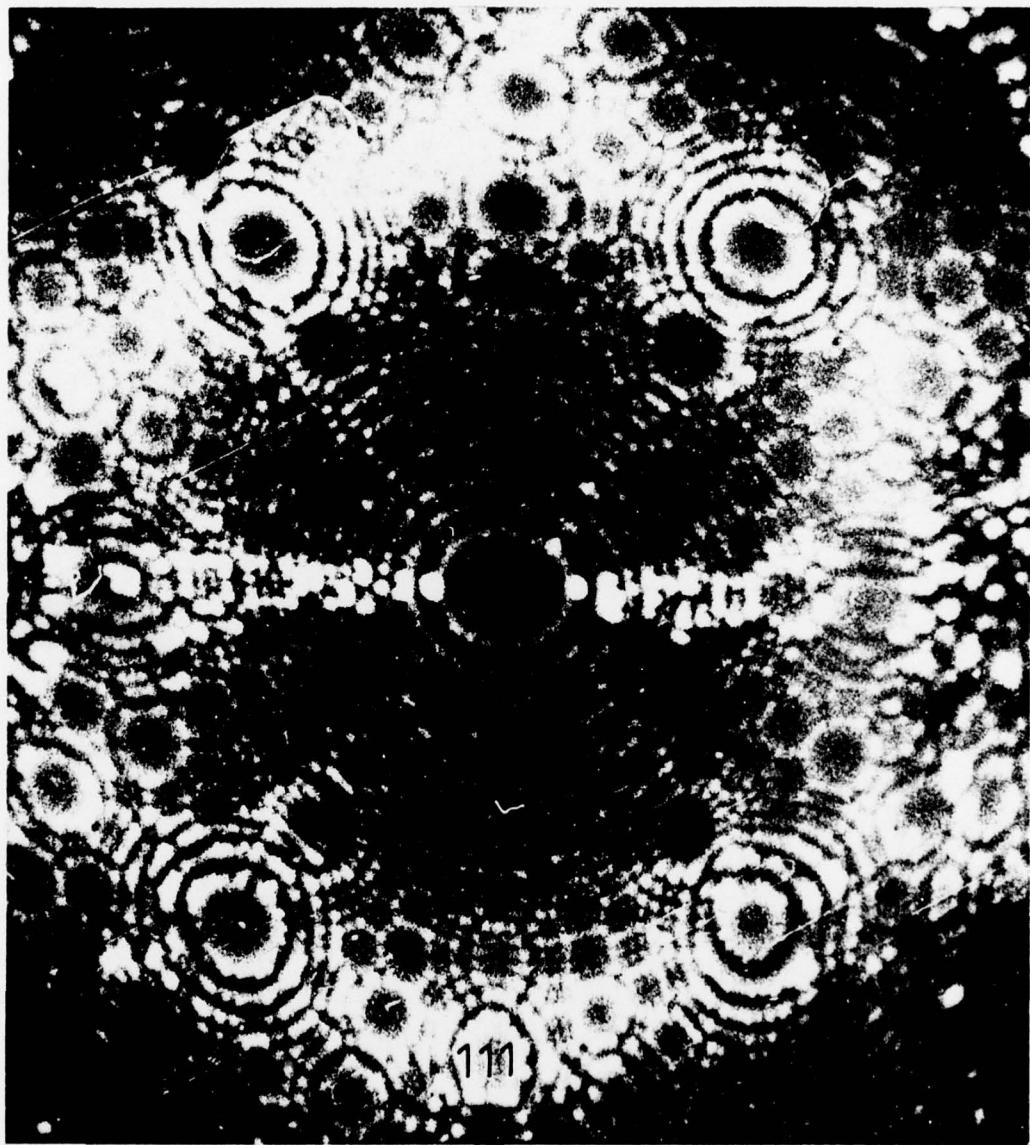


Fig. 10. Tungsten field-ion image showing end-form surface as viewed in the field-ion microscope.

field-ion microscope and associated systems. However, it was not possible during the contract period represented by this report to test the laser system because of problems encountered in the laser power supply and in the laser electronics. The entire system had, however, been made functional at the time this final report was written, and results were being obtained. These results will be reported in conjunction with final report AFOSR 77-3329 to be issued in 1978.

V. SUMMARY

A functional field-ion microscope for in-situ laser irradiation studies has been designed, constructed, and preliminary tests performed during the contract (report) period. Studies of image formation of beryllium in the field-ion microscope have been conducted and beryllium has been found to be unsuited to detailed in-situ laser irradiation studies. Preliminary tests of molybdenum and tungsten have shown that these metals would be more suitable for such studies, and molybdenum would be ideally suited for studies of laser shock phenomena since a detailed study of explosively-induced shock loading in molybdenum has been completed which involved both transmission electron and field-ion microscopy.

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